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TREATMENT OF FINELY DIVIDED PARTICLES AND PARTICLES SO TREATED

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ABSTRACT OF THE DISCLOSURE

Treatment of fillers and pigments to render them hydrophobic and non-structure inducing with a low molecular weight organopolysiloxane in presence of an amino compound, at least part of the process being carried out at an elevated temperature and under pressure.

The invention relates to the treatment of finely divided particles and more particularly to the treatment of fillers, pigments and the like, for example, to render them hydrophobic.

Fillers, pigments and the like materials are used in a wide variety of applications for many purposes in which modification of their surface properties is required including, in some cases, the possession of hydrophobic properties. It is also sometimes desired that fillers or pigments should have reactive groups attached thereto in order to render them more suitable for specific applications.

One of the more important uses of certain of such materials is the incorporation thereof in organopolysiloxane elastomer-forming compositions. One undesirable result of the use of many such materials when used as fillers is that they cause structure build-up and in fact they are commonly known as structure-inducing fillers. Compositions containing these can be formed into the desired final shape only within a comparatively short time if the compositions first undergo a period of reworking, the length of which requires to be increased with the age thereof. This is, of course, highly undesirable and numerous methods have been adopted in order to overcome this defect. Included among such methods has been the treatment of these fillers, pigments and the like with various organopolysiloxanes. The organopolysiloxanes normally used for this purpose have been the lower molecular weight diorganopolysiloxanes, in many cases the cyclic diorganopolysiloxanes such as, for example, octamethylcyclotetrasiloxane which has been widely used for this purpose. The treatment of fillers, pigments and the like with organopolysiloxanes has been carried out by a wide variety of methods. Thus in one method the organopolysiloxane is injected into the finely divided material and allowed to remain in intimate contact therewith until the organopolysiloxane is intimately dispersed through the bulk mass. In another method the vapour of the organopolysiloxanes is passed through a bed of finely divided solid to maintain the solid in fluidised condition, the temperature being sufficient to prevent condensation of the vapour. Excess organopolysiloxane is thereafter eliminated by passing an inert gas through the solid while maintaining it in fluidised condition. In a further method, a finely divided non-alkaline filler or pigment is rendered hydrophobic by treatment with a dihydrocarbylsiloxane of molecular weight not greater than 500 in the presence of from 0.1 to 500 parts by weight per 10,000 parts thereof of an acid. None of the methods proposed or used is, however, entirely satisfactory, particularly where the treated materials are to be used as constituents of organopolysiloxane elastomer-forming compositions.

According to the present invention a new and improved process for rendering finely divided fillers, pigments and like materials hydrophobic and non-structure inducing when used in organopolysiloxane elastomer-forming compositions comprises intimately admixing said material with an organopolysiloxane of molecular weight not more than 500 in the presence of an amino compound, the process being carried out at least in part at an elevated temperature and at least in part under pressure at an elevated temperature.

Fillers, pigments and like materials which may be used in the process of our invention include, for example, the naturally occuring silicas such as diatomaceous earths and ground quartz, manufactured silicas such as precipitated silicas, silica aerogels, fume silicas, for example, the silicas obtained by burning silicon tetrachloride in the vapour phase to give a finely divided silica, asbestos, fullers earth and talc and pigments such as alumina, red iron oxide, magnesium oxide and titanium dioxide.

Fillers, pigments and like materials are normally of particle size not greater than about 10μ although the advantages of our invention can be obtained when the particle size is larger, for example up to 100μ or more. The materials to be treated are generally of relatively large surface area, for example from 20 to 400 m.^{2}/g., but may be of greater surface area, for example up to 1000 m.^{2}/g. The greatest advantage of our invention is obtained in the case of the fillers and especially the finely divided structure inducing silicas, particularly if intended for use as fillers in organopolysiloxane elastomer-forming compositions since structure build-up does not occur until after considerably longer periods than has been the case with the hitherto available treated fillers. The material being treated may or may not contain adsorbed water but in operating our preferred process the presence of adsorbed water is unnecessary.

Organopolysiloxanes suitable for use in the process of our invention include those of molecular weight not greater than 500 and it is in general preferred to use a cyclic diorganopolysiloxane. The organo groups therein, which may or may not be all alike, may be alkyl, alkenyl, aryl, aralkyl, alkaryl, cycloalkyl or cycloalkenyl groups. Suitable groups include, for example, methyl, ethyl, propyl, phenyl, vinyl allyl cyclopentenyl, cyclohexenyl and cyclohexyl groups. It is, however, generally preferred that they be methyl and/or phenyl with or without a proportion of vinyl groups. An especially preferred siloxane is octamethylcyclotetrasiloxane. Suitable siloxanes include, for example, hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, tetramethyltetravinylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethyldisiloxane, sym - tetramethyldivinylsiloxane, sym - trimethyltriphenylcyclotrisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane and other linear diorganopolysiloxanes including hydroxy-ended diorganopolysiloxanes such as 1,7 - dihydroxyoctamethyltetrasiloxane, 1,9 - dihydroxydecamethylpentasiloxane and 1,11 - dihydroxyduodecamethylhexasiloxane. Preferred siloxanes are 1,3, 5,7 - tetramethyl - 1,3,5,7 - tetravinylcyclotetrasiloxane, hexamethyldisiloxane, 1,3 - divinyl - 1,1,3,3 - tetramethyldisiloxane and 1,3,5 - trimethyl - 1,3,5 - triphenylcyclotrisiloxane.

The organopolysiloxane may be used in widely varying amounts, for example, from 1 percent upwards by weight of the material being treated. Normally amounts from about 5 to about 20 percent are adequate for fillers having high surface area, for example, greater than about 200 m.^{2}/g.